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(71) Applicant (for all designated States except US): AHLSTROM MACHINERY OY [FI/FI]; Sentnerikuja 2, FIN-00440 Helsinki (FI).		Published With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments. In English translation (filed in Finnish).	
(72) Inventors; and (75) Inventors/Applicants (for US only): HENRICSON, Kaj [FI/FI]; Munkkiniementie puistotie 12 A 19, FIN-00330 Helsinki (FI). KIISKILÄ, Erkki [FI/FI]; Karhulantie 31, FIN-48600 Karhula (FI).			
(74) Agent: AHLSTROM MACHINERY OY; Patent Dept., P.O. Box 18, FIN-48601 Karhula (FI).			

at least 0.1 mm, preferably 1 - 20 min in order to precipitate metals, especially calcium, present in waste liquor, h) the heat-treated waste liquor flow is passed to further treatment, and i) the pulp from step e) is washed.

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METHOD OF PREVENTING SCALING

The present invention relates to a method of preventing scale formation in a pulping process of chemical pulp.

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Alkaline digestion, sulfate process in particular, has traditionally been carried out by mixing cellulosic material, e.g. wood chips, with cooking liquor into a digestion vessel, the temperature of which is then elevated for a certain period of time to a digestion temperature, typically to 160-180°C, whereby lignin is separated from the wood material and, as a result, chemical pulp and waste liquor are produced. The cooking liquor used in the sulfate process is white liquor containing NaOH and Na₂S. The waste liquor is black liquor containing dissolved lignin, salts of organic acids, extractives, and a significant amount of inorganic compounds.

15 Black liquor is burned in a recovery boiler or other combustion device. As the organic and carbonous compounds in black liquor are combusted, the inorganic compounds in black liquor are transformed into chemicals that can be recirculated and reused in the digestion process. Black liquor is evaporated in a multistage evaporation plant to a higher dry solids content (60 – 90 %) for recovery. In order to recover chemicals and heat the concentrated black liquor is usually combusted in a recovery boiler. The pulp from the digestion, on the other hand, is washed and usually bleached as well for use as raw material for paper.

25 The digestion methods have recently developed enormously and different modifications especially of the sulfate method have been developed. It is typical of these modifications that during the digestion waste liquor is removed from the digester at two or more points of the digestion and correspondingly cooking liquor containing fresh chemical is added. This kind of adjustment of the chemical consistencies in the liquid surrounding the wood material during the digestion has an effect, for example, on the alkali and sulfide profile of the digestion and thus contributes to improving the properties of the pulp produced.

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In addition to lignin, metals that may hamper the treatment of waste liquor after its removal from the digester may also be dissolved from the fibrous material in the cooking liquid during the digestion. Typical metals include calcium, aluminum, iron, magnesium, and copper. Dissolved calcium is likely to form a complex with organic compounds in the digester, and as the waste liquor is evaporated the complex dissociates in the vicinity of a hot heat exchange surface. The released calcium ion then reacts with the carbonate in the liquor, and calcium carbonate forms scales on the heat exchange surface. When the temperature of the waste liquor in the digester has been over 150°C, the complex between calcium and organic compounds dissociates during the digestion and calcium precipitates as carbonate into fibers in the digester and does not usually cause problems in the waste liquor evaporation plant.

Increased scaling in the waste liquor evaporation plant may occur if the waste liquor has been removed from the digester at a temperature below 150°C. In the new digestion methods described above, for instance, the first waste liquor flow may be removed at a temperature below 120°C.

Frederick and Grace (Southern Pulp and Paper Manufacturer 42 (1979) 8:22) have suggested that the formation of calcium scales could be reduced or prevented with a treatment where alkaline waste liquor is heated to a temperature of 150 – 160°C and is maintained at this temperature for 10 – 20 minutes. The deactivation of waste liquor at a temperature of about 150°C has not been widely used since the additional energy it requires may be a considerable investment.

It is an object of the present invention to provide a waste liquor pretreatment method of preventing scale formation, which method can be practiced in the vicinity of the digester with as little additional energy as possible.

The present invention relates to a method of preventing scale formation in a pulping process of chemical pulp, the method comprising at least the following steps:

- a) comminuted cellulosic fibrous material is treated with cooking liquor at a temperature below 120°C,

- b) a first waste liquor flow containing metals, e.g. calcium, is separated from the material and a waste liquor flow is obtained, the temperature of which is below 120°C,
- c) fresh cooking liquor is added to the fibrous material,
- d) fibrous material is digested at a temperature of 140 - 170°C to produce chemical pulp,
- 5 e) a second waste liquor flow is separated from the pulp and a waste liquor flow is obtained, the temperature of which is over 130°C,
- f) the first waste liquor flow from step b) is heated to a temperature over 120°C,
- g) the heated waste liquor flow from step f) is maintained at a temperature over 120°C for at least 0.1 min, preferably 1 - 20 min in order to precipitate metals, especially
- 10 calcium, present in the waste liquor,
- h) the heat-treated waste liquor flow is passed to further treatment, and
- i) the pulp from step e) is washed.

The key concept of the present invention is that by heat-treating those waste liquor flows removed from the digestion, the temperature of which has not risen to over 120°C during the process, scale formation during further treatment of waste liquor can be reduced, during which further treatment chemicals present in waste liquor are recovered and which further treatment typically includes evaporation before combustion in a recovery boiler. The heat-treated first waste liquor flow is not recirculated to the digester. In heat treatment the metal complexes, typically calcium complexes, present in waste liquor dissociate. The released calcium ions react with the carbonate in the liquor forming calcium carbonate crystals that remain in the liquor. The heat treatment is typically carried out so that the liquor is not in contact with the heat exchange surface, but instead, in direct contact with a heat exchange medium such as steam.

25 The present invention is typically applied in connection with continuous digestion, where comminuted cellulosic fibrous material is treated at first with a first cooking liquor. The purpose of this kind of treatment may be, for instance, to provide a high sulfide ion concentration at the beginning of the digestion. The cooking liquor may be white liquor, or waste liquor separated in a later stage of the digestion, which waste liquor still contains significant amounts of digestion chemicals such as NaOH or Na₂S. After the treatment the waste liquor is removed from the digestion at a temperature below 120°C, typically 95 -

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115°C. The amount of this liquor is typically 3 – 6 m³/adt, i.e. 25 – 75 % of the total waste liquor amount to be removed from the digestion.

After the first treatment step, cooking liquor, normally white liquor, is added to the fibrous material and the temperature of the fiber suspension is elevated to 140 – 170°C to produce chemical pulp. A second waste liquor flow, the temperature of which is over 130°C, preferably 140 – 160°C, is removed from the digestion. This waste liquor flow need not be heat-treated, because the metals in it are as compounds in the liquid, e.g. calcium carbonate crystals, that do not form scales during waste liquor evaporation in a multistage evaporation plant of waste liquor.

The first waste liquor flow is deactivated by heating it to over 120°C, preferably to 125 – 145°C, and the liquor is maintained at this temperature for at least 0.1 min, preferably 1 – 20 minutes, whereby calcium carbonate crystals are formed into the liquid. The time is typically at least 0.1 – 5 minutes. The heating is preferably practiced with the heat obtained from the second, hotter waste liquor flow. For this purpose the first and the second waste liquor flow can be mixed with each other. If, after being removed from the digestion, the second waste liquor flow is still used for pretreatment of fibrous material the waste liquor is flashed and the steam produced is brought into direct contact with the first waste liquor flow in order to heat this colder liquor to a desired heat treatment temperature. If the heat energy from the second waste liquor flow alone is insufficient for the heat treatment, the heat treatment may be intensified by introducing additional steam to the heat treatment vessel into direct contact with the waste liquor. Alternatively or additionally, the second waste liquor flow may be heated with steam in a heat exchanger before this liquor flow is used for heating the first liquor flow.

The amount of the first waste liquor to be heat-treated may be reduced by pre-evaporating it before deactivation. The important thing here is that the temperature during evaporation, and especially on the heat exchange surfaces, is so low that the organic complexes of calcium do not dissociate, because that could cause calcium scales on the heat exchange surfaces. The temperature must be lower than the temperature of the waste

liquor when being removed from the digestion. The evaporation temperature is below 110°C, preferably below 90°C, even below 70°C.

5 In evaporation, the amount of the first waste liquor flow can be reduced by over 30 %, even by over 60 %. Thus, the amount of energy needed in the heat treatment diminishes, because there is less liquid to be heated.

10 In a pulp bleach plant, acid effluents are often produced which may also contain high amounts of metals, e.g. calcium, that form complexes with organic compounds. Due to closed water circulations bleaching effluents must also be treated further in the mill. It is often possible to treat bleaching effluents together with waste liquor, whereby, however, the metal compounds in the bleaching effluents may increase scale formation in the waste liquor evaporation plant.

15 In the method according to the invention bleaching effluents may be treated together with the first waste liquor flow in heat treatment. The bleaching effluents may be pre-evaporated with waste liquor before the heat treatment. Here, special care must be taken to ensure that the temperature on the heat exchange surfaces is low. To prevent scaling it may be necessary to treat bleaching effluents at a lower temperature than the first waste liquor. The temperature on the heat exchange surfaces is preferably below 90°C, even below 70°C, after adding bleaching effluents. For this reason it might be necessary to add the bleaching effluents after the waste liquor has been evaporated in one stage at a higher temperature. The boiling point rise of the mixture of the first waste liquor and bleaching effluent is so small that in most cases it is possible to use a two-stage pre-evaporation plant. Thus, the mixture of bleaching effluents and liquor is evaporated at a lower temperature than liquor. On the other hand, bleaching effluents may be evaporated separately before feeding to waste liquor pre-evaporation plant.

30 The normal amount of bleaching effluent introduced from the bleach plant to the treatment of the first waste liquor is 1 – 8 m³/adt, preferably 1 – 5 m³/adt. The temperature is below 100°C, typically 60 – 90°C. The pH is normally 3 – 6.

The invention may be applied in particular in connection with continuous digesters but also with batch digestion having pre-impregnation or pretreatment of the fibrous material at a temperature below 120°C before the digestion.

5 Advantages of this method include:

- An effective way to prevent scaling caused by calcium and equivalent compounds at the evaporation plant.
- The amount of waste liquor to be heat-treated is only about 50 % compared to systems in which the entire waste liquor flow is treated. If waste liquor is pre-evaporated the
10 amount is only about 25 % of the total waste liquor flow.
- The treatment of bleaching effluents may be integrated in the system and thus no separate system is needed for the bleaching effluents. In the method according to the invention, bleaching effluents are simply added to the waste liquor treatment system. This addition enables, for example, the use of a two-stage evaporation plant, which
15 improves the heat economy and reduces the consumption of fresh steam.

In the following the present invention will be described in more detail with reference to the accompanying figures, of which

20 Figure 1 illustrates schematically a preferred embodiment of the invention;
Figure 2 illustrates schematically another preferred embodiment of the invention;
Figure 3 illustrates schematically a third preferred embodiment of the invention; and
Figure 4 illustrates schematically a fourth preferred embodiment of the invention,
disclosing an evaporation plant configuration.

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According to Figure 1 a suspension of comminuted fibrous material, e.g. wood chips, and white liquor is supplied from line 2 to the top of a continuous digester 1. In the first stage in zone I cooking chemicals absorb into the wood material at a temperature of 80 – 120°C, preferably 90 – 105°C. At the same time, alkali of the cooking liquor is
30 consumed, and 15 – 20 % of the wood as well as a considerable part of the metals, especially of calcium, are dissolved.

After zone I, a first waste liquor flow is separated from the fibrous material at screen 3 through line 4. In zone I the treatment temperature is below 120°C, and the temperature of the waste liquor to be removed is typically 95 – 115°C.

5 After this cooking liquor, e.g. white liquor, is added to the fibrous material through line 5. The temperature of the fibrous material suspension is elevated to 140 – 170°C in order to produce chemical pulp. After digestion zone II a second waste liquor flow is separated from the pulp at screen 6 through line 7.

10 The first waste liquor flow 4 may contain, to a detrimental extent, calcium or other metal complexes that are deactivated with the method according to the invention. For this reason the first waste liquor flow is heated to a temperature of over 120°C preferably by means of heat from the second waste liquor flow by mixing the waste liquor flows into line 10. The liquor mixture is maintained at this temperature preferably in a separate
15 retention tank 11 typically for 1 – 20 minutes in order to dissociate the calcium complex and to form calcium carbonate crystals. If necessary, steam can be brought from line 12 into the heat treatment vessel, which steam is then brought into direct contact with the liquor. Alternatively, the second waste liquor flow may be heated with steam in heat exchanger 9 before mixing. The first waste liquor flow cannot usually be heated in an
20 indirect heat exchanger, because that would cause calcium carbonate scales on the heat exchange surfaces.

The heat-treated waste liquor in line 13 is supplied to further treatment through flashing
15 and line 16. Further treatment typically includes evaporation to a high dry solids content (60 – 90 %) for combustion. Turpentine may be separated from the steam
25 obtained from flashing 15, which is one of the advantages of the invention.

The heat economy of the system may be improved by heating white liquor coming in line
17 into digester 1 from causticizing in heat exchanger 14 by means of the heat-treated
30 waste liquor flow 13 or by means of the second waste liquor flow 6 in heat exchanger 8. White liquor is introduced into a desired location in the digester, for example through line 5.

In this embodiment as well as in the ones described later only two removals of waste liquor from the digester are depicted. It is, however, apparent that different digestion modifications may include other liquor removals and liquor circulations as well. For example, there may be more than one first liquor removals to be heat-treated and having at a temperature below 120°C. Also, there may be more than one second, hot (over 130°C) liquor removals. In this figure and also in the subsequent figures there are only two addition points for cooking liquor (WL) although there may be several of them.

10 After zone II the pulp is washed and removed from the digester to washing, screening, and often also to bleaching.

Figure 2 illustrates an embodiment in which the removed waste liquor flows are kept separate and only the first waste liquor flow is heat-treated. The same reference numbers as in Figure 1 are used where applicable with the number "2" added to the front.

A suspension of chips is brought from line 22 to digester 21, and the suspension is treated at the top of the digester with waste liquor removed from the digester in a later step. The liquor still contains significant amounts of cooking chemicals, because the waste liquor has been removed from the digestion in a step where the chemicals have not yet been consumed to a low concentration. At screen 23, a first waste liquor having a temperature below 120°C is separated from the fibrous material to line 24.

After screen 23 cooking liquor from line 25 is added to the fibrous material and the temperature of the suspension is elevated to digestion temperature to produce chemical pulp. After zone II a second waste liquor flow is separated from the pulp to line 27, the temperature of this liquor flow being over 130°C. Since this liquor flow is intended to be returned to the top of the digester to zone I, steam 219 is separated from it by flashing in tank 218 in order to heat the first liquor flow for heat treatment 211. Flashing steam 219 is preferably brought into direct contact with the first liquor flow 24 in a manner known per se. If necessary, additional steam from line 212 may be brought to heat treatment 211 or the second liquor flow may be heated in heat exchanger 29 before flashing.

The heat-treated liquor in line 213 is supplied through flashing 215 and then along line 216 to further treatment. White liquor to be fed to the digester may be heated with the second waste liquor flow either prior to flashing 218 in heat exchanger 28 or after
5 flashing in heat exchanger 221.

Figure 3 illustrates an embodiment in which also the first and second waste liquor flows are kept separate although the reason for keeping the flows separate is not the returning of the second liquor flow to the digester but, for example, the intensifying of the recovery of
10 soap or turpentine. The same reference numbers as in Figure 1 are used where applicable, with the number "3" added to the front.

The first waste liquor flow removed from digester 31 is heated in line 34 with steam obtained from the second waste liquor flow 37 in flashing 318. The flashed second waste
15 liquor flow in line 320 is flashed again in tank 322 and the waste liquor is supplied through line 323 to further treatment.

Figure 4 illustrates an embodiment in which the waste liquor to be heat-treated is pre-evaporated, and the treatment of bleaching effluents is integrated in this process.
20

Cellulosic fibrous material 42 is treated in digester 41 in the same way as described above. The first waste liquor flow in line 44 has a temperature below 120°C and is heat-treated according to the invention. Before this, however, the liquor flow is pre-evaporated in evaporation plant 425, which causes a decrease in the amount of liquid to be fed to heat
25 treatment as well as in the amount of energy needed for the heating.

The amount of energy needed for the pre-evaporation is also small, because the pre-evaporation is carried out at a temperature lower than the temperature at which the first waste liquor is removed, i.e. below 120°C, preferably below 110°C, most preferably
30 below 90°C, even below 70°C. It is important to carry out the pre-evaporation at a temperature as low as possible to prevent calcium or other complexes in the waste liquor from dissociating and forming scales on the evaporation surfaces. The amount of waste

liquor, typically being 3 – 6 m³/adt, may be reduced in pre-evaporation by 30 %, preferably by over 60 %, which means considerable energy savings in view of the heat treatment.

- 5 The treatment of bleaching effluents may be integrated with the treatment of waste liquor. Like the first waste liquor flow, acid bleaching effluents in particular may contain, to a detrimental extent, calcium or other heat sensitive complexes. Bleaching effluents are added from line 424 to the first waste liquor flow, which has the favourable effect of increasing the pH of the effluents. The precipitation of calcium carbonates or other such
10 compounds in heat treatment requires a sufficiently high pH, for example 10 – 12 for calcium carbonate.

- If bleaching effluents are evaporated together with the first waste liquor, it must be ensured that the evaporation temperature is low enough. Bleaching effluents may require
15 a lower temperature, because of higher tendency to form scales. For this reason it may be advisable to add bleaching effluent to the first waste liquor only after the waste liquor has been evaporated in one stage at a higher temperature than is advantageous for the evaporation of bleaching effluent.

- 20 The amount of bleaching effluent to be added to the first waste liquor flow is 1 – 8 m³/adt, preferably 1 – 5 m³/adt. The temperature is below 100°C, typically 70°C. Metal tube evaporators or lamella evaporators are preferably used. Particularly advantageous are falling film evaporators.

- 25 The pre-evaporated mixture of waste liquor and bleaching effluent is passed along line 426 to heat treatment 411. Since the evaporation temperature in the pre-evaporation is low, it is advantageous to preheat the pre-evaporated liquid to a temperature close to that of the first waste liquor removed from the digester, preferably to over 80°C, most preferably to over 100°C. The preheating may be carried out in heat exchanger 427 with
30 the liquor flow to be fed to the pre-evaporation plant. The pre-evaporated and preheated liquor –bleaching effluent mixture is heat-treated in stage 411.

The second waste liquor flow 47 from the digester is flashed as described in connection with the other figures. The flashed second waste liquor flow in line 420 and the heat-treated waste liquor-bleaching effluent mixture in line 426 are evaporated in a conventional multistage evaporation plant 427 of a pulp mill, the evaporation plant
5 typically comprising 5 to 7 stages, and the waste liquor is evaporated to a dry solids content of 60 – 90 %. The first stage of the multistage evaporation plant operates on fresh steam 428, which, if necessary, may also be used in the heat treatment 411. Secondary steam 429 produced in the multistage evaporation plant may, if necessary, be used in the heat treatment 411 for direct steam heating. Secondary steam may also be used in pre-
10 evaporation 425. Steam from flashing 418 of the second waste liquor may be used in the same way. The evaporated liquor is supplied to combustion through line 430.

While the invention has been described here in connection with what is presently considered to be the most practical and preferred embodiment, it will be apparent to those
15 of ordinary skill in the art that many modifications may be made thereof within the scope of protection of the invention, which scope is to be accorded the broadest interpretation of the appended claims so as to encompass all equivalent methods.

Claims:

1. A method of preventing scale formation in a pulping process of chemical pulp, the method , **characterized** in that it comprises at least the following steps:
 - 5 a) comminuted cellulosic fibrous material is treated with cooking liquor at a temperature below 120°C,
 - b) a first waste liquor flow containing metals, e.g. calcium, is separated from the material and a waste liquor flow is obtained, the temperature of which is below 120°C,
 - c) fresh cooking liquor is added to the fibrous material,
 - 10 d) fibrous material is digested at a temperature of 140 - 170°C to produce chemical pulp,
 - e) a second waste liquor flow is separated from the pulp and a waste liquor flow is obtained, the temperature of which is over 130°C,
 - f) the first waste liquor flow from step b) is heated to a temperature over 120°C,
 - g) the heated waste liquor flow from step f) is maintained at a temperature over 120°C
 - 15 for at least 0.1 min, preferably 1 - 20 min in order to precipitate metals, especially calcium, present in the waste liquor,
 - h) the heat-treated waste liquor flow is passed to further treatment, and
 - i) the pulp from step e) is washed.
- 20 2. A method according to claim 1, **characterized** in that in step f) the first waste liquor flow is heated by means of heat from the second waste liquor flow of step e).
3. A method according to claim 2, **characterized** in that in step f) the first and the second waste liquor flow are mixed with each other.
- 25 4. A method according to claim 1 or 2, **characterized** in that the cooking liquor to be added in step a) and/or in step c) is heated with waste liquor from step e) or step g).
5. A method according to claim 2, **characterized** in that the second waste liquor flow from step e) is flashed and the steam produced is used in step f) to heat the first waste liquor flow by bringing the liquor in direct contact with the steam.
- 30

6. A method according to any of the preceding claims, **characterized** in that the temperature of the first waste liquor flow in step b) is 95 – 115°C.
7. A method according to any of the preceding claims, **characterized** in that the temperature of the second waste liquor flow in step e) is 140 – 160°C.
8. A method according to any of the preceding claims, **characterized** in that the first waste liquor flow from step b) is pre-evaporated before step f).
9. A method according to claim 8, **characterized** in that bleaching effluents are added to the first waste liquor flow before evaporation.
10. A method according to claim 8 or 9, **characterized** in that evaporation takes place at a temperature below 120°C, preferably below 110°C, most preferably below 90°C.
11. A method according to claim 1, **characterized** in that bleaching effluents are added to the first waste liquor flow before the heat treatment.

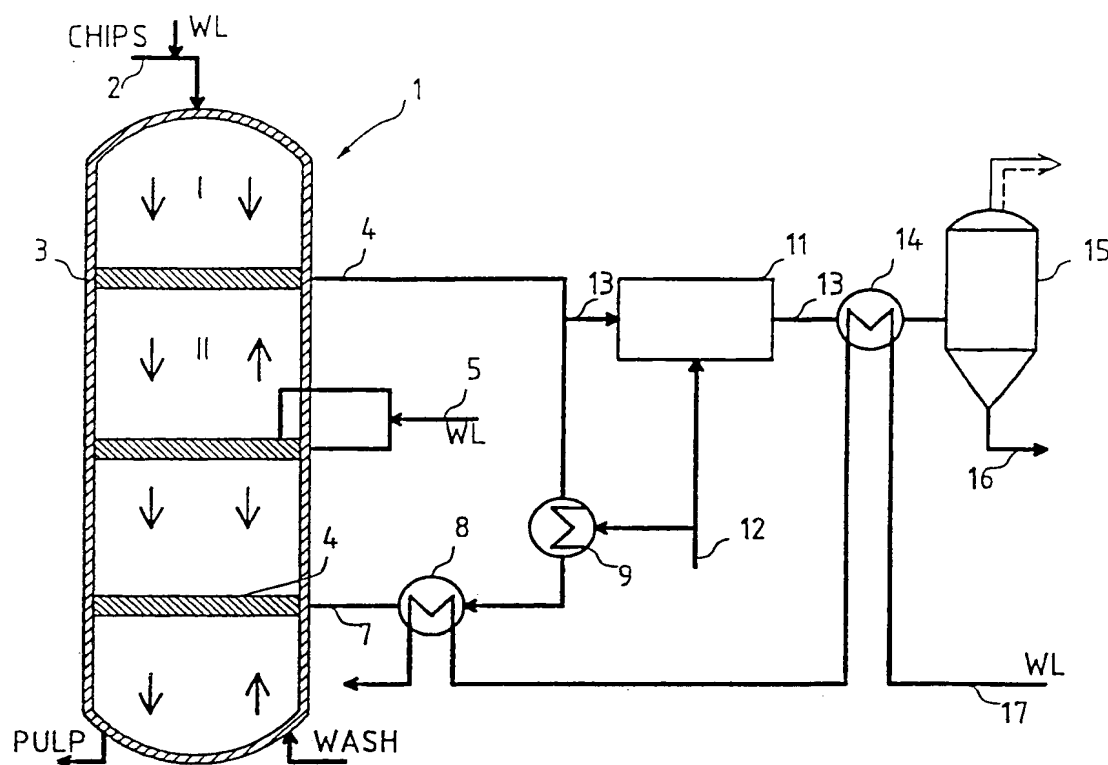


Fig. 1

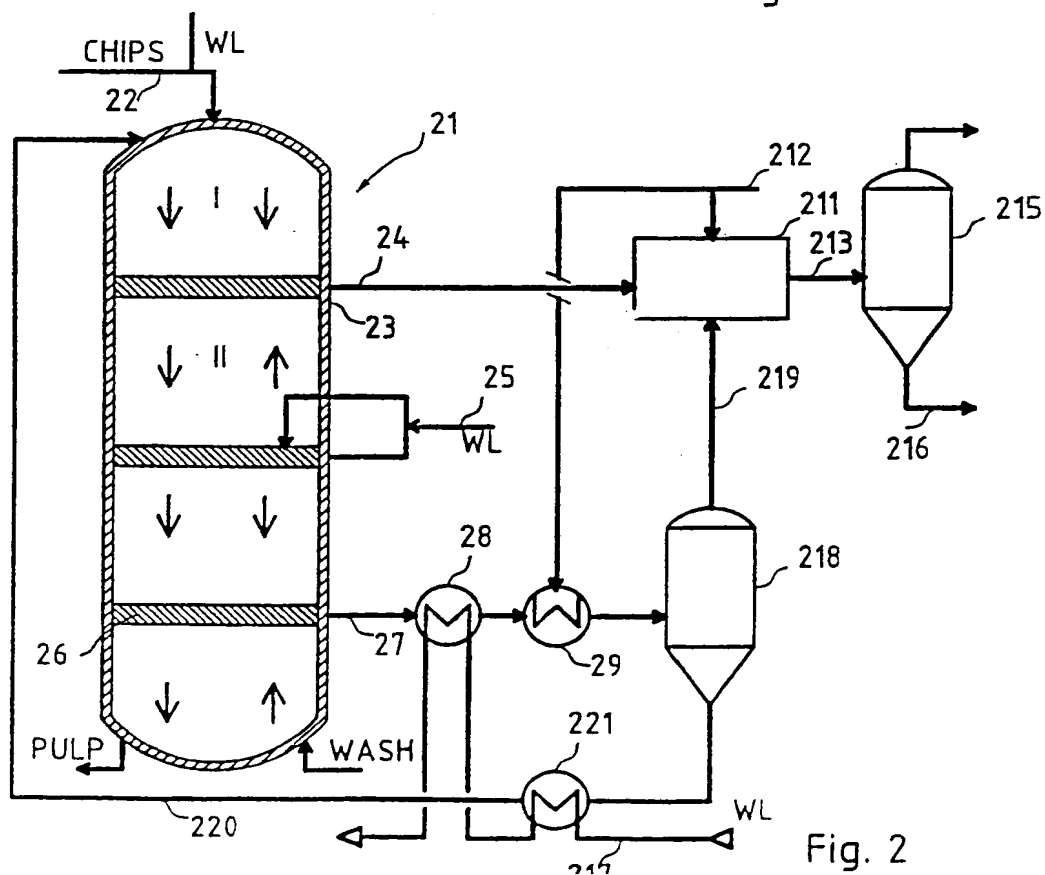
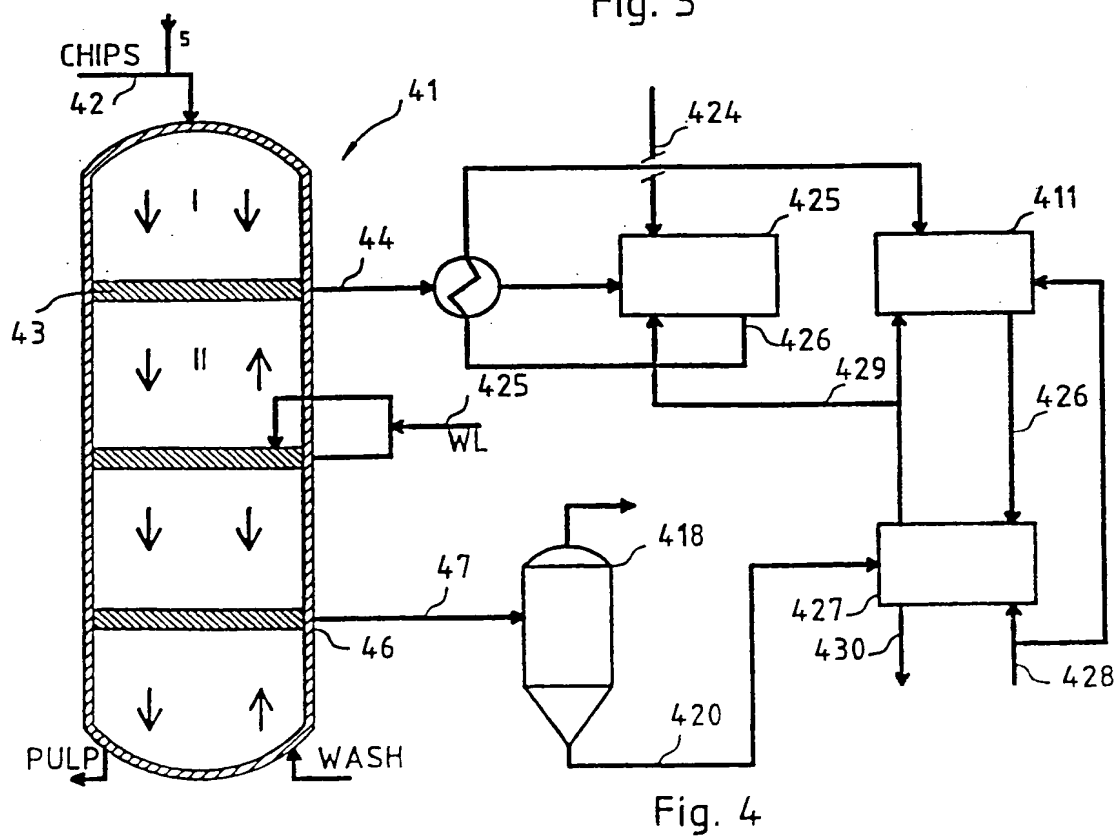
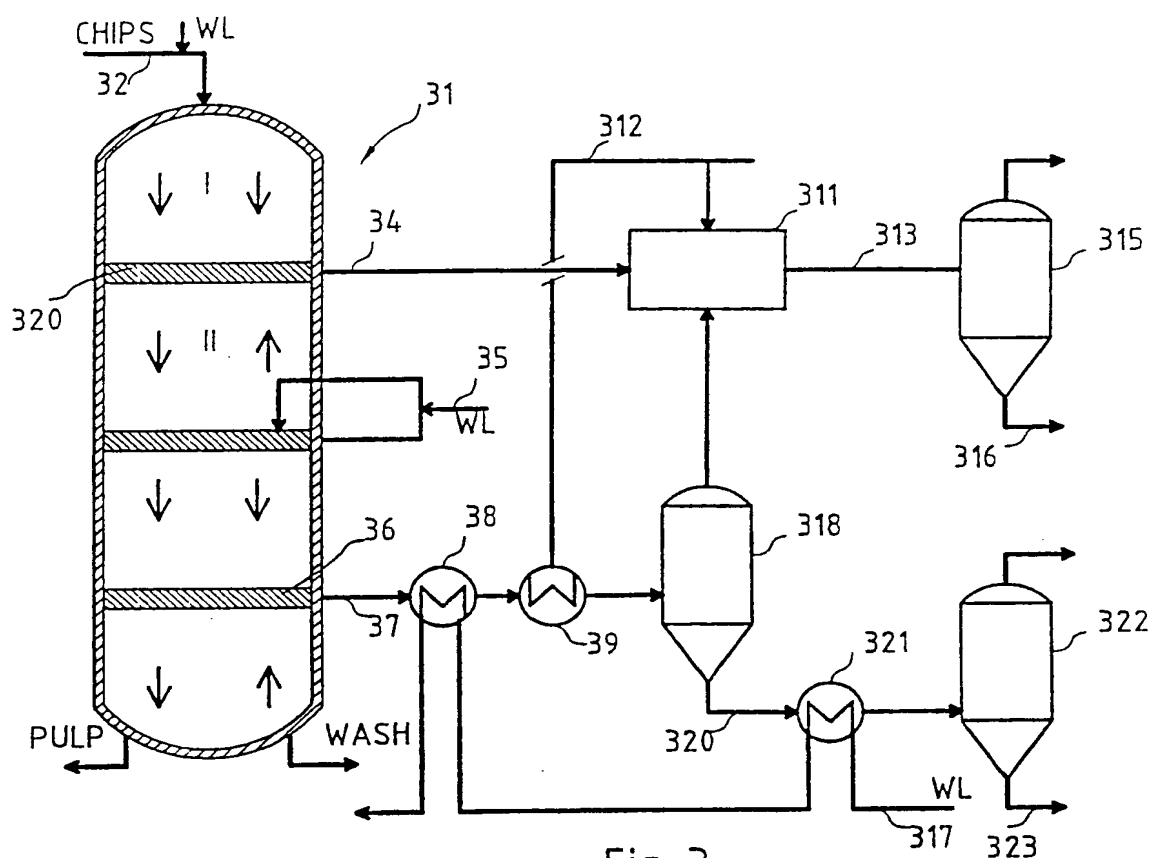


Fig. 2

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 99/00689

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D21C 11/10, D21C 3/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	Southern Pulp and Paper Manufacturer, Volume 40, No 8, August 1977, Thomas M. Grace, "Evaporator Scaling. Research on the Reduction or Elimination of Calcium Scales", page 16 - page 23, figure 8, see especially page 22 --	1,8,10
X	US 4755258 A (ROLF RYHAM), 5 July 1988 (05.07.88), abstract --	1

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 99/00689

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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Information on patent family members

02/12/99

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